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# Determination of Transformation Induced Heat Transfer Coefficients in Mechanical Properties of Powder Metallurgy Steels

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**Abstract:** Powder metallurgy has been used for the manufacture of fully dense, which have not reacted composites consisting of a matrix of copper containing 50 -60 vol% particles with negative thermal ZrW2O8 expansion. On cycling between 25 and 300°C, the compounds showed coefficients thermal expansion which varies rapidly with temperature and significantly larger than expected by theory. Improvements in adhesion strength and thermal properties of composite materials have been achieved in atomized copper alloy with small additions of chromium to increase the interfacial bonding in composite Cu / diamond from a nano-sized thin Cr3C2layer.

**Key words:** Thermal stresses • Heat Treatment • DANTE • Dimensional Residual stresses

### INTRODUCTION

In many applications and areas of powder metallurgy is in direct competition with other manufacturing processes such as casting art of forging or separating techniques Wiedem machining or stamping. For this reason, new methods and processes are developed to enhance the existing To expand application areas and areas and to improve. A possibility of increasing the complexity of powder metallurgical components is in the process combination of powder metallurgy and thixoforging. The powder metallurgy (PM) is concerned with the production of metallic powders and with the production of components made from these powders. Powder metallurgy aluminum and steel components are due to their excellent properties ideal for a variety of industrial applications, especially in automobile (Figure 1) and in household appliances. In the metal industry was the Powder processing used initially in the refractory metals such as tungsten and molybdenum, due to difficult castability of these metals [1]. currently have a powderpressed components due to their different advantages developed wide range of applications in many industries and is becoming increasingly more important. Benefits include a good surface quality, tight weight and dimensional tolerances, excellent material utilization, low Energy consumption during production and very efficient





Fig. 1: Examples of powder metallurgy (PM) -manufactured automotive parts

production of components in large quantities. In certain applications the powder metallurgy has even a technological monopoly such as in the manufacture of hard metals and alloys of metals with different melting points, as well as in the production of materials having a defined porosity. For the production of highly wear-resistant metal-ceramic composites (eg. B. inserts) are powder metallurgical manufacturing process to be essential [2].

Among all the powder metallurgy molding process is the die pressing the economically most significant. The cohesion of the pressed in the form of powder (Green body) is caused by plastic deformation of individual powder grains and their mechanical bonding during compaction. Due to the necessary Pressing process are process engineering sintered components and geometrical Design limits. For example, the maximum size of the components by the construction of the press and the resilience of the pressing tools used limited, whereas the minimum size of the components resulting from the possible volumetric dosage of the powder used. In addition, by demolding the components undercuts and holes that are transverse to the pressing direction, in most cases erstin a downstream step (eg, machining, Drilling) after the pressing and ejecting are introduced. Respect to the geometric design of powder metallurgy components are to avoid be observed by stamp fractures, uneven Dichteverteilungund damage to the green compacts by cracking and spalling sinter fair construction and design guidelines. After the pressing of the components, the sintering of the green bodies is carried out, wherein from the pressed-together particles by a thermally activated mass transport (diffusion) results in a stable compound with a solid structure and composite modified pore geometry. In dependence on the parameter different sintering (sintering temperature, sintering time and sintering atmosphere), the desired material properties such as strength and ductility as well as in certain porosity Setting limits. The sintered components produced in this way have, however, on the basis of their existing residual porosities for certain applications no sufficient strength so that the sintered workpieces to increase the density and strength to be subjected to a post-treatment. For this purpose, the workpieces to be sintered repressed and a second (Zweichfachsintertechnik). The resulting pre-pressing the hardening shall be lifted (recrystallization) and inter-DERS body is again malleable. at Using the same high pressing pressures wiebeim pre-pressing can be explained by the Sizing to achieve a further increase in density and thus the strength. The sinter-forging, however, the advantages of powder metallurgy with the good mechanical properties of forged workpieces Combined. The hardening of the material, the compacting pressure opposes a significant resistance to the re-pressing, which prevents full compression of the material [3]. Applying pressure with simultaneous heating above the recrystallization temperature, this limitation can be overcome [4]. Figure 3 the achievable relative density of the two post-treatment process shown in comparison to the conventional sintering technique. Another process for producing high strength powder metallurgical component is hot isostatic pressing (HIP) is. The main disadvantage of the HIP-method the costs and time involved, since the

duration of the individual process steps can be up to several hours [5]. Other methods make use of the spray forming, in which the powder mixtures in the molten state to a Substrate to be sprayed and then extruded to further compaction or forged [6]. More recent methods utilize the liquid-phase sintering (liquid phase sintering) for the production of sintered components, in which the Powder is partially or fully heated in the liquid phase. This is it, so that the density compared to conventionally sintered materials can be significantly increased for particle rearrangement in the sintered body.

Literature Survey: The ability of a material under a shearing stress function of time to produce a decrease in viscosity up again gradually to "regenerate" once the load is removed. Non-eutectic alloys show in the temperature range between solidus and Liquidus thixotropic deformation behavior. DieFormgebung of mtallischen alloys in this temperature range is therefore as Thixoforming referred to. In thixoforming at the time of shaping, a certain percentage of the material or already fixed and the rest in liquid form. The thixotropic area for steel materials between the solidus and liquidus in the iron-carbon diagram highlighted. Above the liquidus line, the process of casting and aufder other side borders (below the solidus) on the process of forging [7]. A material in the thixotropic state exposed to a shear stress, there is a drastic decrease of the viscosity, a high fluidity of the material, which leads. When there is no load it comes to re-increase of the viscosity. The processing of steel and other high-melting alloys by However, the method provides Thixoformings thixoforming of low melting far greater demands on systems engineering and Prozessführungals Light metal alloys. Diehohen forming temperatures sometimes exceeding 1400 °C make special demands on all involved steps [8], but especially the type and design of the shaping tools. The advantages of this technology are, however, in comparison to the forging in the production of complex Geometries in a single forming step and compared to the casting at elevated Cycle times and reduced shrinkage of the components. In the 1990s, studies were conducted to thixotropic forming, although these are mainly concerned about serving. As a model material came because of their comparatively low Melting point and large solidification interval often speed steels like. In addition to high-speed steels were basic research on Thixoformbarkeit high-alloy steels subject of several works in which simple model components were produced [9-11]. In more recent work, the investigations were extended to low-alloy steels such as the tempered steel

C55 and the bearing steel 100Cr6. moreover There are studies on the thixotropic molding of other materials such as Aluminum, copper and titanium alloys, as well as Composites. In Figure Samples thixogeschmiedete steel components from different research projects are presented. Production of complex geometries of particle-reinforced steel materials to powder by pressing with subsequent thixoforging at the Institute for Metal Forming and Metal Forming (IFUM) conducted basic research be where the advantage Eder two methods of powder metallurgy and thixoforging be combined. This novel process combination to the existing design limits, resulting from the presented design guidelines for sintered components, extended and highly wear-resistant components are produced by the addition of ceramic particles. Due to the low viscosity during forming, also can produce complex parts in one forming step. In the work presented here are preliminary investigations at the scheduled Presented process chain, beidenen samples from two different steel powders are pressed, sintered and then compressed isothermally in the thixotropic area. For the manufacture of steel pressings Magnetic iron powder (MIP), the graphite (Carbon) was added and used a high-alloy steel powder. Both powders are produced via the powder metallurgical route by water atomization and have a nodular grain shape [12]. In Figure 7, the Controls and particle distribution are shown for the steel powders used. Both powders are characterized by a minimum oxygen content and have a high carbon content of about 2% (MIP-powder) and 1% (6-5-3 HS powder). the HS 6-5-3 powder has gegenüberdem MIP powder has a large number of other alloying elements and can be of the group of alloyed tool steels assign or high-speed steels (HSS). Both powders also have a relatively similar size distribution, whereinsaid maximum particle sizes in With approximately 250 microns and the HS 6-5-3 powder indicated MIP powder with approximately 150 microns [13]. To estimate the thixotropic area of the two materials a DSC Analysis performed at a heating rate of 10 K/min under protection gas argon and the start and end points of the phase transition determined. This is the MIP powder. Within a range of about 1160-1410 °C and the HS 6-5-3 powder between about 1230-1430°C. Figure 8 shows the recorded DSC curve is for the MIP-powder and determined thixotropic area shown. The values determined indicate that the material on a relatively slow increase in the liquid phase fraction within the thixotropic interval out. In contrast, HS 6-5-3 powder is a steeper Increase to identify the beginning of the thixotropic area. The steel powder are two-sided uniaxial pressing at a pressure compacting of 500 Mpa with the addition of 2 wt%.- Aramidwachs. each sample each has a weight of 12 g and a diameter of  $\emptyset = 12$  mm. as a result the different compressibility of the two metal powders have the MIP samples a height of about 17.1 mm whereas the HS 6-5-3 samples have 18.1 mm. This corresponds to a relative density of 6.2g/cm and 5.9g/cm. Subsequently, the pellets over a period of 60 minutes at a Temperature of 1100°C under vacuum sintered [14].

### MATERIALS AND METHODS

Thermal aspects are becoming increasingly important for the reliability of electronic components due to the continuous advancement of the electronics industry. Therefore, effective thermal management is a key issue for the packaging of high-performance semiconductors [15]. the ideal material working as a heat sink and heat spreader should have a coefficient of thermal expansion (CTE) and a high thermal conductivity. Metal matrix composites (MMC) offer the possibility to adapt the properties of a metal by adding an adequate reinforcing phase to meet the needs in thermal management. When the diamond particles are embedded in a copper matrix, the interface plays a crucial role in determining the thermal conductivity, CTE and also the mechanical properties of the composite. An ideal interface should provide a good grip and minimal thermal boundary resistance [16]. Copper pure liquid does not wet diamond and Cu / diamond composites made from powder metallurgy have shown to characterize weak interfacial bonding; debonding occurred on thermal cycling. It is well known that the copper alloy with a strong carbide forming element promotes wetting and bonding of diamond. Even in the case of binding solid phase (for example, hot pressing), high bonding strength was observed for copper alloys with small additions of Ti, Cr, B or Zr. The influence of interface formation on the thermal conductivity of these compounds is not yet fully understood [17]. Electrons dominate heat conduction in copper, while phonons that dominate the diamond. So, for the thermal conductivity of the MMC, the energy transfer must occur between electrons and phonons. It is assumed that a very thin layer of interface of a carbide phase can help the necessary electron-phonon coupling. To solve the problem of the interface between copper and diamond, the use of different carbide forming alloying elements added as the matrix of copper was investigated. High thermal conductivity were made with diamond-reinforced matrix composites CuCr or CUB. Quick pressure assisted sintering techniques with rates of heating / cooling of 100-150 K stero<sup>-1</sup> and taking TimesUp few minutes to produce the most promising thermal conductivity values han the conventional hotpressing with rates of heating / cooling of 10 K min<sup>-1</sup> [18]. In the present work, the interface characteristics of highly conductive CuCr / diamond composites have been studied in detail, because the morphology of the interface and thermophysical properties depend on the sintering technology. A CuCr 0.8 powder pre-alloyed (by weight.%) Prepared by gas atomization was used to study the effect of adding this alloying element to the matrix Cu. The average particle size (d50) of this powder is in the range 10-15m. The chromium content of selected corresponds to the optimum content of alloy required for a strong bond matrix / diamond composites in CuCr / diamond [19]. The reinforcement used is a synthetic diamond grit of 70/80 mesh, to a particle diameter in the range corresponding 177-210µm. The synthetic (IB)-type MBD4 diamond was bought in Luoyang High-Tech Qiming Superhard Materials Co. Ltd, Luoyang, Henan, China. The thermal conductivity of intrinsic type IB single crystals depends strongly impurities and was shown correlation with the concentration of nitrogen present in the diamond lattice. A nitrogen content of 200 ppm was measured for the diamond powder delivery for hot extraction with LECO O / TC 600 Nanalyser Therefore a conductivity of diamond 1500 W (mK)<sup>-1</sup> vial of being engaged in this work. The apparent density of the composite was measured using a method based on the law of Archimedes' and compared to the theoretical density. It was found that the relative density of the composites is 98%. The measurement of the CTE was performed in a Netzsch dilatometer to the following conditions: temperature range 25-300°C; heating rate of 5 K min<sup>-1</sup>; argon atmosphere. The thermal conductivity was measured by pyrometry Xenon with nanoFlash from Netzsch, Germany. Fracture surfaces of composite samples were analyzed by scanning electron microscopy (SEM) using equipment from Zeiss EVO 50 [20]. Furthermore, the diffraction patterns recorded by a Siemens D5000 diffractometer were used to study the interfacial phases

## **RESULTS**

The samples are in each case and for forming in a thermal container at different temperatures in a temperature interval between 1000°C Heated 1280°C and then in a servohydraulic Umformsimulator (Plastometer) compressed. The degree of deformation was uniformly



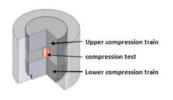


Fig. 2: Servo hydraulic forming simulation (left) and thermal containers (on the right)

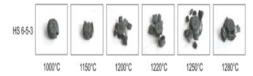


Fig. 3: Free upsetting the HS 6-5-3 samples

 $\square$  = 0.7 at a strain rate of  $\square$  = 1/s. The Plastometer used and the thermal container used with the integrated edging paths shown. Addition was carried out on all samples, respectively upsetting a powder compact only heated to the respective temperature (without conversion), so that the respective structural state before and after the conversion or the influence of temperature can be studied specifically.

After forming and heating the samples from the containers were thermo removed and swapped in the air. The transformed samples from the two steel powders are shown. A visual comparison of the two Materials can already be applied to a highly different material behavior during include forming. DieHS 6-5-3 samples have at 1000°C and 1150°C. significant defects in the form of cracks in the outer region. The composite material Here, however, remains largely. It was not until 1200°C occurs a complete separation or flaking of material regions, so that the larger sample in a Number of parts crumbled. At 1280°C, however, no clear separation of smaller areas more material to detect, but the material forms a almost contiguous composite material. This is due to the increased proportion of liquid phase due within the sample, which exerts a cohesive or adhesive effect between the powder particles.

The samples from the MIP-powder have, however, after the forming process at 1000°C. bursting on the border areas, where no complete separation of the material structure can be seen. Between 1150°C and 1250°C show the MIP rehearse macroscopic cracks in the edge region whereas both upset Sample at 1280 °C only a very small number of micro-cracks in the outer region to is seen. This ductile material behavior could also be attributed to



Fig. 4: Free upsetting the MIP samples

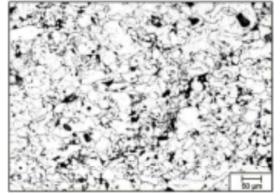


Fig. 5: Sintered Micrographs of the samples heated HS 6-5-3

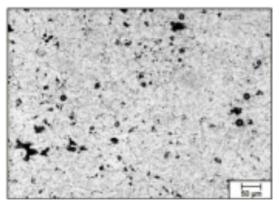


Fig. 6: Sintered (1200°C) Micrographs of the samples heated HS 6-5-3

increased Its share in the liquid phase due within the test specimen, which can reduce a normal appearane and the further spread of cracks.

In a subsequently performed metallographic examination can be detect significant differences in the material structure. For this, each of the created both for the central area as well as for the heated sintered and reshaped powder samples micrographs. In the HS 6-5-3 samples are in the exclusively sintered compact yet clearly identify the boundaries between the individual powder grains. The addition to the samples after sintering Wrought or Thixotemperatur were heated in contrast, have a function the temperature used a more homogeneous microstructure on at the only partially the (original) grain boundaries are visible.

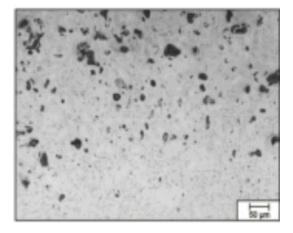


Fig. 7: Sintered (1200°C) Micrographs of the samples heated HS 6-5-3

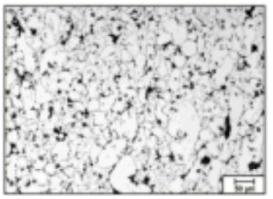


Fig. 8: Sintered Micrographs of the deformed samples MIP

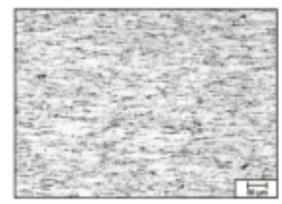


Fig. 9: Sintered + deformed (1000°C) Micrographs of the deformed samples MIP

In the MIP samples are also a similar effect in the deformed samples can be observed. Again, the structure function of the deformation temperature significant differences in the compound of powder grain receiving. At 1000°C is also an anisotropic orientation of the material

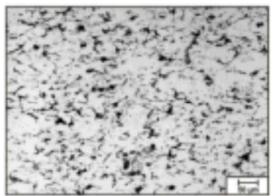


Fig. 10: Sintered + deformed (1200°C) Micrographs of the deformed samples MIP

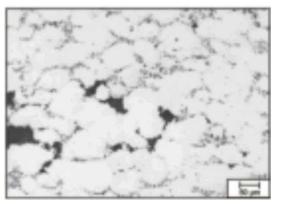


Fig. 11: Sintered + deformed (1280°C) Micrographs of the deformed samples MIP

structure as a result of the compression process visible. This characteristic microstructure is however at 1200°C and 1280°C no longer discern what is possibly due to strong recrystallization effects and the partial occurrence of the liquid phase in the material.

This should be investigated to what extent reduce the use of these methods or a combination of the sintering process can fully substitute. Furthermore offers the powder metallurgical route Opportunity to reinforce only partial areas with ceramic particles. in this Manner can be produced specially adapted to the case of load components.

#### CONCLUSION

Three processing routes have been developed to fabricate composite materials Cu/ZrW<sub>2</sub>O<sub>8</sub> with 50 vol% ceramic -60 and without reaction with little or no residual porosity of: (I) the high-pressure, low-temperature hot-pressing the mixed metal oxide ceramic powder; (ii) Low-pressure, high-temperature, hot isostatic pressing

coppercoated ceramic powder; and (iii) the high pressure, low temperature, hot-pressing the mechanically alloyed metal-ceramic powder. The thermal expansion of the composites was measured between 25 and 300°C. The first heat cycle (and to a lesser extent the subsequent thermal cycles) showed hysteresis, time-dependent recovery, as well as coefficient of thermal expansion significantly greater than that predicted from the composite elastic theory, these side effects were reduced in mechanically alloyed composites. This anomalous behavior can be explained by the reversible allotropic transformation of ZrW<sub>2</sub>O<sub>8</sub> between its high-pressure ã-phase and its lowpressure á-or \( \beta \) ZrW<sub>2</sub>O<sub>8</sub> phases, accompanied by a substantial volume change. Experimental calorimetry and diffraction measurements and theoretical stress estimates show that ã- ZrW<sub>2</sub>O<sub>8</sub> is first used by the high isostatic pressure during processing, formed. During the subsequent heating and cooling hydrostatic tensions produced in the particles by the thermal expansion difference between matrix and amplification are sufficient to induce the reversible allotropic transformation. To the best of our knowledge, This is the first time that a phase transition was observed in a metal matrix composite material as a result of thermal mismatch stresses.

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